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# प्लास्टिक – परीक्षण पद्धतियाँ

## भाग 3 भौतिक तथा आयामीय गुणधर्म

## अनुभाग 9 फास्फोरस पेन्टाआक्साईड के क्लोमैट्रिक पुनरुत्पादन द्वारा प्लास्टिक में नमी ज्ञात करना

*Indian Standard*

# PLASTICS — METHODS OF TESTING

## PART 3 PHYSICAL AND DIMENSIONAL PROPERTIES

## Section 9 Determination of Moisture in Plastics by Coulometric Regeneration of Phosphorus Pentoxide

ICS 83.080; 19.060

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 3

Methods of Test for Plastics Sectional Committee, PCD 23

## FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards after the draft finalized by the Methods of Test for Plastics Sectional Committee, had been approved by the Petroleum, Coal and Related Products Division Council.

Moisture can seriously effect the processability of plastics. High moisture content may cause surface imperfections (that is, splay or bubbling) or degradation by hydrolysis. Low moisture (with high temperature) may cause solid phase polymerization.

The physical properties of plastics may be greatly affected by the moisture content.

Polymers, such as nylon 6, that contain high-boiling materials such as plasticizers or high levels of monomer may contaminate the electrolytic cell. This will cause the cell to become unresponsive, requiring it to be cleaned and recoated. The directions for cleaning and recoating the electrolyte cell are given in Annex A for guidance.

While preparing this standard considerable assistance has been derived from ASTM D 4019 : 1994 a 'Standard test method for moisture in plastics by coulimetric regeneration of phosphorus pentoxide' issued by American Society for Testing and Materials (ASTM), USA.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

# *Indian Standard*

# PLASTICS — METHODS OF TESTING

## PART 3 PHYSICAL AND DIMENSIONAL PROPERTIES

## Section 9 Determination of Moisture in Plastics by Coulometric Regeneration of Phosphorus Pentoxide

### 1 SCOPE

**1.1** This standard prescribes a test method which is used for the determination of moisture in most plastics. Plastics containing volatile components, such as monomers and plasticizers, may contaminate the electrolytic cell, used in the test, causing incorrect results.

**1.2** This test method is suitable for measuring moisture over the range from 0.005 percent to almost 100 percent.

### 2 NORMATIVE REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

| *IS No.* | *Title* |
|---|---|
| 170 : 1986 | Acetone (*third revision*) |
| 1070 : 1992 | Reagent grade water (*third revision*) |

### 3 APPARATUS

**3.1 Moisture Analyzer**

**3.1.1** An oven capable of heating the sample to at least 250°C.

**3.1.2** A bypass valve system for the saturation prevention of electrolytic cell, needed on one type of instrument.

**3.1.3** An electrolytic cell for trapping and electrolyzing the moisture.

**3.1.4** Electronics for converting the electrolysis current to digital data.

**3.1.5** Digital display for presenting the digital data as micrograms of water.

**3.2 Sample Holders,** made from nickel, stainless steel, glass, or other nonreactive material.

### 4 REAGENTS

**4.1 Purity of Reagents**

Reagent-grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

**4.2 Purity of Water**

Unless otherwise indicated, reference to water shall be understood to mean grade 3 reagent water, as defined in IS 1070.

**4.3 Acetone** (*see* IS 170)

**4.4 Nitrogen,** less than 5 µg/g (5 ppm) water.

NOTE — The nitrogen carrier gas shall be supplied to the instrument through metallic, fluorocarbon, or other moisture-impermeable tubing. Use of other tubing may not permit the instrument to be properly zeroed because of atmospheric moisture permeating into the instruments.

**4.5 Phosphoric Acid,** 85 percent.

**4.6 Sodium Tartrate**

**4.7 Potassium Tartrate**

**4.8 Sodium Tungstate**

**4.9 Nitric Acid**

### 5 TEST SPECIMEN AND SAMPLE

**5.1** Samples may be in the form of moulding powder, fabricated shapes, or moulded items.

**5.2** Specimens may be cut from fabricated shapes or moulded items.

**5.3** Since the specimen size is small, 10 g or less (*see* **7.4.2**), care shall be exercised to ensure that the specimen is representative of the sample.

**5.4** Hygroscopic resin samples shall be protected from the atmosphere.

### 6 CALIBRATION AND STANDARDIZATION

**6.1** The calibration factor is determined by using materials with known water content.

**6.2** Four methods are described for use with the calibration materials:

- i) Water;
- ii) Sodium tartrate dehydrate;
- iii) Sodium tungstate dehydrate, and
- iv) Potassium tartrate.

The recommended calibration material is sodium tungstate dehydrate, which is stable (in the anhydrous form) to temperatures above 1 000°C. Tartrates can decompose above 220°C and can cause deterioration of the detector.

### 6.3 Method for Use with Water

A glass capillary (micropipette) is used to measure a known mass of water, typically 2 mg.

**6.3.1** Prepare analyzer for use as described in 7.

**6.3.2** Fill the micropipette by holding it at midpoint with a pair of tweezers and dipping lightly in distilled water.

**6.3.3** Remove the moisture from the sides of the pipette with absorbent tissue. Take care not to touch the end of the pipette with the tissue because it will absorb the moisture from the capillary.

**6.3.4** After filling the pipette, place it in the sample holder and set the holder in the oven.

**6.3.5** Run the analysis and record the water counter reading as standard reading ($R_{std}$). Use an analysis time of 20 minutes and a temperature of 150°C.

### 6.4 Method for Use with Sodium Tartrate Dehydrate

This method uses sodium tartrate dehydrate with a theoretical water content of 15.66 percent as the basis of standardization.

**6.4.1** Prepare the analyzer for use as described in 7.

**6.4.2** Weigh to the nearest 0.1 mg, 10 to 13 mg of sodium tartrate dehydrate and place in the sample holder.

**6.4.3** Place sample holder and contents in the oven.

**6.4.4** Repeat as described in **6.3.5**.

### 6.5 Method for Use with Sodium Tungstate Dehydrate

This method uses sodium tungstate with a theoretical water content of 10.92 percent as the basis of standardization.

Repeat the steps given in **6.4.1, 6.4.2, 6.4.3**, and **6.4.4** except use 15 to 19 mg.

### 6.6 Method for Use with Potassium Tartrate Hemihydrate

This method uses potassium tartrate hemihydrate with a theoretical water content of 3.83 percent as the basis of standardization.

Repeat the steps given in **6.4.1, 6.4.2, 6.4.3**, and **6.4.4** except use 25 to 30 mg.

**6.7** Calculate the calibration factor, *K*, in micrograms per microgram reading, as follows:

$$K = \frac{F \times W_{std}\ 1\,000}{(R_{std} - R_{B})}$$

where

$K$ = calibration factor in micrograms per microgram (µg/µg);

$F$ = fraction of standard material attributed to water:

water method $F = 1.00$

sodium tartrate method $F = 0.156\,6$

sodium tugnstate method $F = 0.109\,2$

$W_{std}$ = mass of standard material, mg;

$R_{std}$ = reading for standard material, µg; and

$R_{B}$ = reading for blank, µg.

**6.8** The calibration factor should be 1.00 ± 0.10. A factor outside these limits would indicate a problem such as a contaminated or deteriorated detector.

## 7 PROCEDURE

### 7.1 Preparation of Sample Holders

**7.1.1** If boats are used, dry as many boats as convenient at 100 to 105°C in a circulating air oven for at least 1 h.

**7.1.2** Remove the boats from the oven and immediately place them in a desiccator to cool for at least 15 min before using.

**7.1.3** If boats are not used, then store the sample holder within the oven.

### 7.2 Initial Settings

**7.2.1** Adjust the source nitrogen pressure to 35 ± 3 kPa.

**7.2.2** Adjust the nitrogen flow control valve to a flow rate of 70 ± 20 cm$^3$/minute. This adjustment shall be made with the sample oven closed.

**7.2.3** Adjust the analyzer background count rate by setting the microgram water content to a counting rate of 0.8 ± 0.3 µg/minute for the DuPont instrument.

**7.2.4** Adjust the oven temperature control to the required test temperature for the plastic being analyzed.

NOTE — The material specification shall be checked for the required test temperature. If the test temperature is unknown, it will have to be determined experimentally (*see* Annex B).

### 7.3 Determination of Blank

**7.3.1** Open the analyzer oven and set a dried sample holder ready for insertion.

**7.3.2** Expose the holder and analyzer oven to the laboratory atmosphere for 20 ± 3 seconds. Place the sample holder in oven and close. The timing period starts at the instant the oven door is opened and ends when it is closed.

**7.3.3** Reset the water counter to zero.

**7.3.4** Adjust the time control to the required test time for the plastic being analyzed.

**7.3.5** At the end of the required test time, record the water counter reading as the blank, $R_B$.

NOTE — For long test times, the time required to determine the blank ($R_B$) may be determined by determining the background count rate ($C$) and the accumulated background ($B$) after 10 min have elapsed, then multiplying this count rate by remaining time and adding it to the accumulated background:

$$R_B = B_{10} + C\,(t - 10\text{ min})$$

where

$R_B$ = reading for blank, µg

$B_{10}$ = accumulated background after 10 minutes, µg

$C$ = background count rate, µg/minute; and

$t$ = total analysis time, minutes

### 7.4 Determination of Sample Moisture

**7.4.1** Remove the sample holder from oven.

**7.4.2** Place appropriate sample size in the sample holder (*see* Note). The holder shall be filled and returned to the oven within 20 ± 3 seconds.

NOTE — Sample size is based on the expected moisture content of the sample. The following table may be used as a guide:

| *Expected moisture Content* (percent) | *Approximate Sample Size,* (g) |
|---|---|
| 0.005 to 0.1 | 2 |
| 0.1 to 0.5 | 0.5 |
| 0.5 to 1.0 | 0.2 |
| 1 to 5 | 0.05 |
| 5 to 10 | 0.02 |

**7.4.3** Return the sample holder to the analyzer oven.

**7.4.4** Adjust the time control to the required test time.

**7.4.5** At the end of the required test time, record the water counter reading as the sample reading, $R_B$.

**7.4.6** Reset the water counter to zero.

**7.4.7** Remove the sample holder from the oven.

**7.4.8** When the sample has cooled, remove it from the sample holder and weigh it to ± 0.1 mg, $M$ (sample).

NOTE — This method has been written for materials that do not adhere to the sample holder. Materials that do adhere shall be weighed before placing in the sample holder.

## 8 CALCULATION

$$\text{Moisture content, percent} = \frac{K(R_S\ R_B) \times 0.1}{M}$$

where

$R_S$ = reading for sample, µg;

$R_B$ = reading for blank, µg;

$K$ = calibration factor reading, µg/µg (shall be near unity) (**6.7**); and

$M$ = mass of the sample in mg.

## 9 REPORT

The test report shall include the following information:

a) Complete identification of the sample tested including type of material, source, manufacturer's code and form,

b) Date of test,

c) Individual specimen readings,

d) Individual specimen blanks readings,

e) Individual specimen moisture readings,

f) Individual specimen moisture content, and

g) Average moisture content.

# ANNEX A

(*Foreword* and *Clause* B-1.3)

## DIRECTIONS FOR CLEANING AND RECOATING THE ELECTROLYTIC CELL

**A-1** Remove the electrolytic cell from the instrument.

**A-1.1** Connect one port of the cell to a source of vacuum such as a (water) aspirator or vacuum pump with suction flask.

**A-1.2** Flush the cell with distilled water at least five times, to remove the old coating.

**A-1.3** Flush the cell with acetone at least five times.

**A-1.4** Dry the cell for 2 min by aspirating air through the cell.

**A-1.5** Reinstall the cell in the instrument and establish nitrogen flow. The uncoated cell should not cause the count to increase. If it does not increase, go to step **A-1.3**. If it does increase, further cleaning of the cell is required, proceed with step **A-1.6**.

**A-1.6** Fill the cell with concentrated nitric acid and let it stand for half an hour.

**A-1.7** Drain the acid, flush the cell several times with distilled water, followed by acetone and aspirate dry.

**A-1.8** Again, remove the electrolytic cell from the instrument and apply vacuum to one port.

**A-1.9** Prepare a 2 to 8 solution by volume of reagent grade (85 percent) phosphoric acid in acetone.

**A-1.10** Fill the cell with the phosphoric acid solution. Withdraw the excess solution.

NOTE — Do not flush the cell with excess phosphoric acid solution, which may cause a build-up of phosphoric acid and deactivate the cell.

**A-1.11** Dry the cell for 2 min by aspirating air through the cell.

**A-1.12** Reinstall the cell in the instrument and electrolyte the cell by purging it with nitrogen for at least 30 min before turning on the timer.

NOTE — The cell may be electrolyzed outside the instrument by applying a 65-V dc to the electrodes while purging the cell with nitrogen.

**A-1.13** When the system is sufficiently dry so that it may be balanced, proceed with instrument calibration (*see* **6**).

# ANNEX B

(*Clause* 7.2.4)

## SELECTION OF TEST TEMPERATURE AND TIME

**B-1** Selection of the proper test temperature and time is often a compromise between cell life (that is, time until recoating is necessary) and time of analysis. High temperatures may degrade the sample and evolve monomer or plasticizer causing loss of sensitivity. Low temperatures reduce the evolution of moisture and prolong the analysis. Selection of the optimum test temperature and time is, therefore, a trial-and-error process.

**B-1.1** Select the specimen size to contain about 2 mg of moisture and set the temperature at 110°C.

**B-1.2** Analyze the sample until the count rate reduces to a level where analyzing the sample for less than an additional minute changes the resultant count by <1 percent. If the total time is less than 20 min, the time and temperature are satisfactory. If the time is greater than 30 minutes, repeat steps **B-1.1** and **B-1.2** at a temperature 10°C higher.

**B-1.3** If, after making several tests under the conditions determined in **B-1.1** and **B-1.2**, loss of sensitivity is observed, clean and recoat the cell (*see* Annex A) and lower the temperature of future tests by 10°C.

NOTE — Suggested test conditions for selected plastics are given in Annex C.

# ANNEX C

(*Clause* B-1.3)

## SELECTED TEST CONDITIONS

**C-1** Suggested Test Condtions for Selected Plastics are given in Table 1.

**Table 1 Suggested Test Conditions for Selected Plastics**

| Sl No. | Plastics | Temperature (°C) | Test Time (Minutes) | Sample Size (g) |
|---|---|---|---|---|
| (1) | (2) | (3) | (4) | (5) |
| i) | Polyamide | 175 | 30 | 0.2 |
| ii) | Thermoplastic polyester | 150 | 45 | 1 |
| iii) | Ionomer | 150 | 30 | 1 |
| iv) | Polyolefin | 150 | 20 | 2 |
| v) | Polycarbonate | 180 | 20 | 2 |
| vi) | ABS | 130 | 20 | 0.2 |

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 323 01 31, 323 33 75, 323 94 02

Telegrams: Manaksanstha
(Common to all offices)

Regional Offices:

| | | Telephone |
|---|---|---|
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| Eastern | : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Maniktola<br>CALCUTTA 700054 | 337 84 99, 337 85 61<br>337 86 26, 337 91 20 |
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